TEXAS
Biosolids Quarterly

a Texas Natural Resource Conservation Commission Publication PD-013/96-4

This volume covers the sewage sludge and soil sampling requirements of 30 Texas Administrative Code (TAC) Chapter 312. This volume also gives an overview of sampling procedures, the methods required by the rule to analyze samples, and a list of publications that provide detailed information about sewage sludge and soil sampling and analysis.

Sewage Sludge Sampling

Chapter 312 requires the sampling and analysis of sewage sludge for certain inorganic pollutants, pathogens, and vector attraction reduction if the sewage sludge is land-applied, placed on a surface disposal site, or incinerated. The rule does not provide specific instructions on how to sample; however, the rule prescribes the frequency for monitoring and lists the analytical methods that must be used to analyze different type of samples.

Who Must Sample?

In most cases, the preparer of sewage sludge (usually the owner or operator of the treatment works) will be responsible for sampling the sludge for metals, pathogens, and, where applicable, for vector attraction reduction. Often the generator is also the one who prepares, land-applies, surface-disposes of, or incinerates the sewage sludge. Sometimes a person other than the generator is the preparer (a person who provides additional processing that may alter the quality of sewage sludge before its use or disposal). That preparer may also be required to sample the additionally processed sewage sludge before it is subjected to land application, surface disposal, or incineration. The owner / operator of a surface disposal site is also responsible for sampling metals under certain circumstances, that is, when needed to meet site-specific limit requirements and / or when the boundary of an active biosolids unit is less than 150 meters from the property line of the surface disposal site (EPA, 1994).

How Often Should Sampling (Monitoring) Be Done?

The minimum monitoring frequencies for sewage sludge that will be subjected to land application, surface disposal, or incineration are listed in 30 TAC Chapter 312. The frequency of monitoring depends on the amount of sewage sludge used or disposed of annually. The required monitoring frequency increases with the amount of sewage sludge used or disposed of. Monitoring must take place at least as often as indicated in the table below to demonstrate compliance with 30 TAC Chapter 312 pollutant limits and pathogen and vector attraction reduction requirements.

Amount of sewage sludge* *(metric tons per 365-day period)*	**Frequency**
Greater than zero but less than 290	Once per year
At least 290 but less than 1,500	Once per quarter (four times a year)
At least 1,500 but less than 15,000	Once per 60 days (six times a year)
At least 15,000	Once per month (12 times a year)

** Amount of sewage sludge (other than domestic septage) land-applied, placed on an active biosolids unit, or fired in an incinerator; dry weight basis.*

The monitoring frequency should anticipate the potential for changes in metals concentration, pathogen density, and vector attractiveness in sewage sludge. Monitoring frequency also should take into account when sewage sludge is actually being used or disposed of. The rule assumes, especially in regard to preparers of large amounts of sewage sludge, that the sewage sludge will be used or disposed of consistently throughout the year. If sewage sludge is being stored for a number of months before use or disposal, a large mass could accrue. Although the rule does not require analysis until the biosolids are used or disposed of, the preparer, land-applier, or disposer might want to take composite samples for analysis throughout the storage period so that sampling results are more representative and the operation affords better process control (EPA, 1994).

In this issue...

When Should Samples Be Taken?

Sewage sludge must meet the requirements of the regulations at the time of use or disposal or at the time it is prepared, if distributed in bags for marketing to the general public. Sampling and analysis should take place before use or disposal of the sludge so that analytical results can be available ahead of time. Sewage sludge could be sampled and analyzed for metals content a considerable period of time before its use or disposal, provided that no significant additional reduction in volatile solids content has occurred. Certain pathogen and vector attraction reduction determinations, however, would need to be made close to the time of use or disposal to meet the requirements of the rule. In some cases, sampling may need to be conducted over the applicable period of time to show that reduction of parameters has been achieved (EPA, 1994). When the level of one or more pollutants or pathogens in the sewage sludge being tested is close to the regulatory limits, or if there is a high potential for spikes, then analyzing ahead of time can avoid exceeding regulatory limits.

Where Should Samples Be Collected?

In general, samples should be collected while the sewage sludge being sampled is in motion rather than stationary. The movement of sewage sludge helps in achieving better mixing and thus a more uniform entrainment of solids and pollutants. Liquid sludges should generally be sampled from pipelines or preflushed pipeline ports far downstream in the treatment works. When sampling liquid sludges from lagoons, include

continued

the floating, suspended, and sediment layers of the sewage sludge. A more representative sample of dewatered sludge can be obtained by sampling from moving conveyor belts or front-end loaders that are moving a pile of sewage sludge. If the sewage sludge sample must be taken with the sewage sludge in place, random samples from the entire area should be taken and combined. In most cases, sewage sludge is sampled at the end of the treatment process, just prior to its use or disposal (EPA, 1994).

What Types of Sampling Equipment Should Be Used?

Sampling equipment (e.g., coring devices, coliwasas, pitchers, conduits, shovels, trowels, containers) must be made of materials that will not contaminate or react with the sewage sludge. Suitable sampling equipment materials generally include glass, stainless steel, and plastic (Teflon, polyethylene, polypropylene). Any steel equipment used must not be galvanized or zinc-coated, because such a coating will contaminate the sample. Moreover, all equipment should be kept clean to avoid contamination. For samples used to demonstrate compliance with Class A pathogen requirements, sampling equipment should be sterilized prior to sampling (EPA, 1994).

What Size of Sample, Sample Equipment, and Storage Times Should Be Used?

It is important both to collect the correct amount of sewage sludge needed to perform sample analysis and to preserve and store samples properly. Appropriate containers, sample sizes, preservation, and storage times for sampling sewage sludge for metals and pathogens are given below.

Metals (solids and semisolids):

Container:	Plastic or glass (wide-mouth container)
Preservation means:	Store cool (4°C)
Maximum storage time:	6 months
Minimum sample volume:	300 mL

Metals (liquids):

Container:	Plastic or glass (wide-mouth container)
Preservation means:	Add nitric acid (HNO_3) to pH < 2
Maximum storage time:	6 months; mercury (Hg) only 28 days
Minimum sample volume:	1,000 mL

Pathogens and Vector Attraction Reduction (biological):

Container:	Sterile plastic or glass (wide-mouth container)
Preservation means:	Store below 4°C
Maximum storage time:	Varies; analyze as soon as possible.
Minimum sample volume:	1-4 liters

Soil Sampling

Chapter 312 requires the sampling and analysis of soil for certain inorganic pollutants, nutrients, salinity, and soil pH if the sewage sludge is land-applied or placed on a surface disposal site. The analysis of soil samples is necessary to evaluate residual nutrient supplies and to formulate fertilizer recommendations. Soil testing is essential in planning and designing good crop and sludge management programs. Soil testing is also needed to obtain information on the background concentrations of the metals existing at the site. *The most important part of a good soil testing program is getting a good sample of soil to test.*

Who Must Sample?

Any person or entity who wishes to obtain a registration for the beneficial use of sewage sludge or a permit for the surface disposal of sewage sludge from the TNRCC is required to submit a detailed soil analysis report. Also, any person or entity who has a registered beneficial use site is required to submit annually a soil analysis report to demonstrate that the waste is used properly and is applied at agronomically sound rates.

When Should Samples Be Taken?

Initial soil samples required during the application process for beneficial land use registration may be taken whenever necessary. Samples submitted annually to fulfill the reporting requirement for a current registration must be taken within the same 45-day time frame each year. In general, fall is the best time to collect soil samples (take soil samples before any application of commercial fertilizer, manure, or other substances).

Where Should Samples Be Collected?

Soil samples should truly represent the field on which sludge will be applied, since a good sample is vital for getting good soil test information. If the field is quite uniform, one composite sample for each soil depth per 80 acres is sufficient. Avoid sampling areas such as dead furrows or back furrows, lime, sludge, or manure piles, animal droppings, fences or roads, rows where fertilizer has been banded, eroded knolls, and low spots. If a field contains two or more distinctly different kinds of soils, take separate samples of each soil, so as to obtain at least one composite sample for 80 acres. An alternate soil sampling plan is allowed, provided the plan is approved by the TNRCC prior to the taking of any samples.

Composite Sample Preparation:

Collect at least 10-15 random sample cores from each of two soil depth zones – 0-6 inches and 6-24 inches. Mix the various cores or slices together in a clean plastic container, and take a subsample to be put into the sample bag.

How Often Should Sampling (Monitoring) Be Done?

Chapter 312 requires annual soil sampling to obtain analytical results showing the concentration of nutrients, salinity, and soil pH. The soil-sewage sludge mixture shall be resampled for inorganic pollutants (metals) at 4.5 years after beneficial use site registration issuance.

What Types of Sampling Equipment Should be Used?

Any one of several different tools, such as an auger, soil sampling tube, or spade, may be used. Sample tubes or augers should be either stainless steel or chrome-plated. Sampling tools should be kept clean to avoid contamination. If using a pail to collect the soil, it should be plastic to avoid cross-contamination from trace elements (e.g., zinc).

Spades and Scoops:

The simplest and most direct method of collecting samples is with the use of a normal lawn or garden spade and a stainless steel scoop. This method can be used in most soil types. Very accurate and representative samples can be collected with this procedure.

Hand Augers:

A hand auger system consists of an auger bit followed by a bucket type of cylinder. A "T" handle is employed with various lengths of extensions to obtain samples from various depths. Hand augers remove six-inch increments of soil from the bore hole. This is very effective when samples need to be obtained at different locations in one bore hole.

What Size of Sample, Sample Equipment, and Storage Times Should Be Used?

Appropriate containers, sample sizes, preservation, and storage times for sampling soil for metals and nutrients are given below.

Container: Paper bags lined with plastic, plastic or glass bottles

Preservation means: Soil samples should be air-dried (at a temperature below 40°C) and ground as soon as possible after collection. Chemical analyses are generally performed on air-dried samples, which do not require special preservation for most parameters. Samples collected for nitrate and ammonia analyses should be refrigerated under moist field conditions and analyzed as soon as possible.

Minimum sample volume: 100 grams

Sewage Sludge and Soil Sample Analysis

What Must Be Sampled?

Sewage Sludge:

Regulated parameters include inorganic pollutants (metals), pathogens, vector attraction reduction characteristics, nutrients, and emissions (sewage sludge incinerator emissions only). Parameters to be analyzed depend on final use or disposal.

Land Application:

- Metals (arsenic, cadmium, chromium, copper, lead, mercury, molybdenum, nickel, selenium, and zinc)
- Pathogen and vector attraction reduction
- Nutrients (total nitrogen, ammonium nitrogen, nitrate-nitrogen, total phosphorus, and total potassium)

Surface Disposal:

- Metals (arsenic, cadmium, chromium, copper, lead, mercury, molybdenum, nickel, selenium, and zinc)
- Pathogen and vector attraction reduction
- Methane gas in air

Incineration:

- Metals (arsenic, cadmium, chromium, copper, lead, mercury, molybdenum, nickel, selenium, and zinc)
- Incinerator emissions (total hydrocarbons or carbon monoxide, oxygen, temperature, information needed to determine moisture content, and mercury and beryllium, when applicable)

Soil:

Soil depth 0-6": Nitrate-nitrogen, ammonia-nitrogen, total nitrogen, phosphorus, potassium, sodium, magnesium, calcium, soluble salts, soil water pH, aluminum, arsenic, cadmium, chromium, copper, lead, mercury, molybdenum, nickel, selenium, zinc

Soil depth 6-24": Nitrate-nitrogen, ammonia-nitrogen, total nitrogen, phosphorus, soil water pH

What Methods Should Be Used to Analyze Samples?

The following methods, other methods as approved by the executive director of the TNRCC, or the latest revision shall be used for analyzing samples:

- Enteric viruses should be quantified using ASTM D 4994-89 as described in Section 11 of *Standard Practice for Recovery of Viruses from Wastewater Sludges, Annual Book of ASTM Standards.* Water and Environmental Technology, ASTM, Philadelphia, PA, 1992.
- Fecal coliform should be quantified by method described in Part 9221 E or Part 922 D, *Standard Methods for the Examination of Water and Wastewater,* 18th edition, American Public Health Association, Washington, D.C., 1992.
- Helminth ova should be quantified by method described in Yanko, W.A., *Occurrence of Pathogens in Distribution and Marketing Municipal Sledges,* EPA / 600 / 1-87 / 014, 1987.
- Salmonella sp. bacteria should be quantified by method described in Part 9260 D, *Standard Methods for Examination of Water and Wastewater,* 18th Edition, American Public Health Association, Washington, D.C., 1992, or in Kenner, B. A. and H. P. Clark, "Detection and enumeration of Salmonella and *Pseudomonas aeruginosa," J. Water Pollution Control Federation,* Vol. 46, No. 9, pp. 2163-2171, 1974.
- Specific oxygen uptake rate should be quantified by method described in Part 2710 B, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, American Public Health Association, Washington, D.C., 1992.
- Inorganic pollutants should be quantified using the appropriate EPA method prescribed in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* EPA Publication SW-846, 3rd edition (1986) with Revision I.
- Total, fixed, and volatile solids should be quantified by method described in Part 2540 G, *Standard Methods for the Examination of Water and Wastewater,* 18th Edition, American Public Health Association, Washington, D.C., 1992.
- Percent Volatile Solids Reduction Calculation should be quantified by method described in the guidance document *"Environmental Regulations and Technology - Control of Pathogens and Vectors in Sewage Sludge,* EPA / 625 / R-92 / 013, U.S. Environmental Protection Agency, Cincinnati, OH, 1992.
- Total nitrogen should be quantified using Kjeldahl digestion or

continued

an equivalent accepted procedure. The use of methods that rely on mercury as a catalyst is not acceptable.

- ▲ Electrical conductivity should be determined from an extract of a 2:1 (volume:volume) water-soil mixture.
- ▲ Soil pH should be analyzed by the electrometric method in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, EPA Publication SW-846, 3rd edition (1986) with Revision I.

Documentation

Sampling Plan

It is recommended that each organization have a sampling plan that identifies the sampling locations, sample volume, days and times of collection, required equipment, preservation techniques, instructions for labeling samples and ensuring chain of custody, and a list of contact persons and telephone numbers in case unexpected difficulties arise during sampling.

Sampling Log

It is essential that all information pertinent to a sampling event be recorded in ink in a hardbound notebook with numbered pages. This information is very helpful when a sampling situation has to be reconstructed, without depending on the collector's memory. Suggested entries in the log book include, at a minimum:

- ▲ time, date, and weather conditions;
- ▲ purpose of sampling;
- ▲ location of sampling;
- ▲ collection method;
- ▲ name and address of the field contact;
- ▲ number and volume of samples taken;
- ▲ description of sampling points;
- ▲ sample preservation method used in the field;
- ▲ type and size of sample container.

Chain of Custody

The chain of custody is a form that documents the history of the sample from the time the sample is collected to the time it is analyzed in the laboratory. This information is needed to prove that the samples are handled and transported in a manner that preserves the integrity of the sample. This record is particularly important if the sample is to be introduced as evidence in litigation. The chain of custody should at least contain:

- ▲ the collector's name;
- ▲ the signature of collector;
- ▲ date and time of collection;
- ▲ place and address of collection;
- ▲ signatures of all persons involved in the chain of possession.

References

1. *A Plain English Guide to the EPA Part 503 Sewage Sludge Rule.* U.S. EPA, Office of Wastewater Management, EPA / 832 / R-93 / 003, September 1994.
2. *Environmental Regulations and Technology: Control of Pathogens and Vector Attraction in Sewage Sludge.* U.S. EPA, Office of Research and Development, Cincinnati, OH. EPA / 625 / R-92 / 013, December 1992.
3. *POTW Sludge Sampling and Analysis Guidance Document.* U.S. EPA, Permits Division, Washington, D.C. First edition, August 1989.
4. *Guide to Soil Suitability and Site Selection for Beneficial Use of Sewage Sludge.* Oregon State University Extension Service, Manual 8, March 1990.

FOR MORE INFORMATION regarding sewage sludge and soil sampling, please contact the TNRCC Sludge and Transporter Review Team at (512) 239-4710.

Watershed Management Division, MC 148
Texas Natural Resource Conservation Commission
P.O. Box 13087
Austin, TX 78711-3087

BULK RATE
U.S. POSTAGE PAID
AUSTIN, TEXAS
PERMIT NO. 1967